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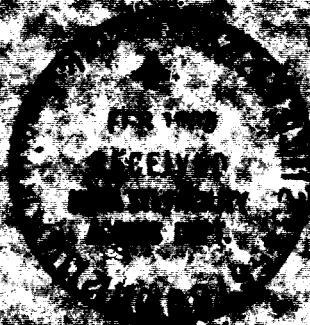
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SURFACE PROFILE CHANGES OF SCUFFED BEARING SURFACES

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ABSTRACT

A phase-locked interference microscope capable of resolving depth differences to 30 Å and planar displacements of 6000 Å was constructed for the examination of the profiles of bearing surfaces without physical contact. This instrument was used to determine surface chemical reactivity by applying a drop of dilute alcoholic hydrochloric acid and measuring the profile of the solid surface before and after application of this probe. Scuffed bearing surfaces reacted much faster than unscuffed ones, but bearing surfaces which had been previously exposed to lubricants containing an organic chloride reacted much more slowly. In a separate series of experiments, a number of stainless steel plates were heated in a nitrogen atmosphere to different temperatures and their reactivity examined later at room temperature. The change of surface contour as a result of the probe reaction followed an Arrhenius-type relation with respect to heat treatment temperature. This result could have implications on the scuffing mechanism.

INTRODUCTION

When nonconforming solid surfaces separated by a lubricant are in relative motion under increasing loads, the bulk temperature gradually increases and the lubricant film thickness decreases. At some point the surfaces will begin to interact mechanically. At first, the peaks of the highest asperities will be reduced and the surfaces will become smoother. This is an essential part of normal "run-in." At higher loads scoring occurs manifested by grooves and ridges in the surface oriented in the sliding direction. At still higher loads a sudden failure may occur, which is called scuffing. The scoring and scuffing events are often coincident and many authors use these terms interchangeably.

Because scuffing is likely to be catastrophic and come without warning, measures were designed to postpone or avoid it. Since welding of the contacting surfaces is considered an essential part of scuffing, these measures usually entail the formation of interposed layers. For example, extreme pressure (E.P.) lubricant additives are thought to cause formation of renewable coatings of metal sulfides, chlorides, or phosphates preventing the welding. Durable surface coatings consisting of thin, hard, inert layers such as

titanium nitride could also avert scuffing for the same reason. These coatings may even function under starved or unlubricated conditions.

Although the mechanism of scuffing is not fully understood, a temperature criterion is commonly postulated. The best known theory is the flash temperature concept of Blok [1], which consists of the bulk temperature of the metal surface and the instantaneous temperature rise in the contact area. Others correlated scuffing failure primarily with chemical activity which, of course, is strongly influenced by temperature (Goldman [2]). Implicitly, the assumption has been made that, in the presence of reactive species, chemical reaction takes place at or above some characteristic (total) contact temperature. Another possibility is that portions of metal surfaces exposed to higher temperatures may become inherently more reactive at lower temperatures due to metallurgical or chemical changes.

Recently a phase-locked interference microscope was developed [3] which is capable of measuring vertical displacements to 3×10^{-9} m (30 Å) and horizontal displacements to 6×10^{-7} m (6000 Å). Minute changes in surface topography, which are the result of surface chemical reactions, can be measured with this apparatus. Obviously, changes in surface reactivity may also be determined.

Therefore, the objective of this investigation is to determine the differences in chemical reactivity of unscuffed and scuffed ball bearing surfaces by measuring changes in surface topography caused by an acid etch of alcoholic hydrochloric acid. Both uncoated and titanium-nitride-coated stainless steel (440 C) ball bearings were studied. Results were compared with scanning electron micrographs.

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APPARATUS

Phase-Locked Interference Microscope

The first instrument of this kind was originally designed and built for the examination of optical surfaces by Johnson, Leiner, and Moore [3] at the Institute of Optics at the University of Rochester. The instrument is a Twyman-Green interferometer with alternating current electronics, which is capable of a phase resolution of one two-hundredth of the wavelength of the red He-Ne laser line (6.328×10^{-7} m) (6328 Å). Surface profiles can thus be obtained to about $\pm 3 \times 10^{-9}$ m (± 30 Å) without physical contact. Figure 1 is a schematic drawing of the instrument's optics. Radiation from a He-Ne laser line is split by a beamsplitter where part of the radiation continues to a flat mirror oscillated by a piezoceramic transducer, and part is deflected to the sample surface by a microscope objective. The split beams are reflected back to the beamsplitter, recombined, expanded by a microscope objective, and ultimately transferred to a photoelectric detector. Two scanning mirrors allow scans parallel to the x and y directions in the sample plane. Phase differences between the two beams generate fringes on the detector plane. As the sample surface is scanned, the phase difference between the two beams changes, and the fringes are moved over the detector surface proportionally as determined by the optics.

The sensitivity of the instrument is derived from an ingenious electronic arrangement. The reference mirror is oscillated piezoelectrically at 20 kHz. Correspondingly, the fringe pattern at the detector surface is oscillated with the same frequency, but the intensity of a fringe peak or valley is oscillated at twice the reference mirror frequency or at 40 kHz. Intuitively this arises from the fact that the peak, or valley intensity, and the corresponding photoelectric potential can change in one direction only--downward for the peak and upward for the valley--while the radiation intensity anywhere else can become stronger or weaker. It is thus possible to "lock in" a peak at the 40-kHz frequency. The mean potential (halfway between peak and valley) or dc potential will follow the locked-in fringe and become a measure of the phase change as the sample surface is scanned.

Distance calibration of the instrument is simple. In the horizontal plane the distances of successive peaks of a reflection grating provide the required information. Depth calibration is done very accurately by the phase jump in the detector plane. The separation between two successive interference fringes corresponds to a phase difference of 2π between the sample and reference beams. In air, because of the phase reversal on reflection by the sample surface, this separation of fringes corresponds to a change in depth of sample surface profile of half a wavelength of the laser light 3.164×10^{-7} m (3164 Å). In terms of the electric recorder output potential this geometrical change corresponds to 10 V. Any voltage changes are directly proportional to the phase changes, and therefore to the changes of depth, and the conversion can be carried out in terms of the 316.4-Å/V relation.

However, changes may occur in the optical properties or surfaces which in turn may affect the output from the interference microscope. These changes may be brought about by oxidation, heating, wear, or other chemical reactions. Therefore care must be taken in interpreting the surface profile changes obtained with this instrument.

Ball/Plate Sliding Contact

The experimental setups used to obtain scuffing or scoring were described in an earlier publication [4]. These were the ball-plate sliding elastohydrodynamics (EHD) contacts in which a bearing ball of 5×10^{-7} m (2.25 in.) diameter was turned by a horizontal shaft either on top of a diamond window (the plate) in the bottom of a cup containing the test fluid or underneath a sapphire window (the plate). In either case the ball was loaded from the top. The former setup is used primarily for infrared analysis of the fluid under EHD conditions; the contact is always flooded. The latter setup is used for EHD film thickness and traction measurements. Conditions were such that oil starvation did not occur.

MATERIALS

The bearing balls were 5.7×10^{-2} m (2.25 in.) in diameter and made of 440 C stainless steel. Some were coated with titanium nitride by chemical vapor deposition (CVD) to a uniform thickness of about 4 μ m, as described by Hinterman and Boving [5]. The initial smoothness of all ball surfaces was about 0.01 μ m (mean peak height).

The balls were run with polyphenyl ether lubricant (5P4E) either pure or containing 1 percent by volume of 1,1,2-trichloroethane. The stainless steel reference plates were of smoothness similar to that of the balls. They were made of polished 304 stainless steel and were 25 by 25 by 0.4 mm in size. The acid etch was 0.04 M hydrochloric acid in absolute ethyl alcohol.

EXPERIMENTAL PROCEDURES

Heating of Reference Plates

The reference plates were sandwiched in a holder between half-inch-thick asbestos plates containing a 1.9×10^{-2} m (0.75 in.) diameter hole in the center. Two pieces of a material melting or changing color at known temperatures were clamped on the top of the sample, allowing temperature monitoring during heating. The two temperature indicators were selected so that the maximum heating temperature was bracketed between them.

Heating was accomplished rapidly (at ~ 220 K/s) with the hot gases just above the tip of an acetylene flame directed at the lower side of the plates. At the same time, and for a considerable time thereafter, nitrogen gas was blown over the top of the plate to reduce oxidation (cooling rate: ~ 100 K/s). Oxidation could never be entirely prevented because of residual adsorbed oxygen.

Preparation of Ball Samples

The balls were taken from the EHD rigs after many hours of operation using polyphenyl ether with and without 1 percent by volume of 1,1,2-trichloroethane. The balls were cleaned with acetone, soaked in absolute ethyl alcohol overnight, rinsed, and placed in a vacuum desiccator to remove the adsorbed alcohol.

Interference Microscope Examination of Ball Surfaces Run in Polyphenyl Ether

Since the score or scuff marks on the uncoated balls were typically 100 μ m in width, it was useful to work with two different microscope objectives, 10X and 40X--the former enabling us to overlap the mark and the latter to look inside it.

Figure 2 shows two areas on a used bearing ball, which includes parts within and outside the scuff mark. A drop (~ 0.05 ml) of 0.4 M alcoholic hydrochloric acid was applied from a measuring syringe, with care being taken not to move the ball-microscope alignment in any way. For this work the ball was rigidly mounted on a holder, which itself was rigidly attached to the microscope frame, so that the entire system would move as one unit. As soon as the drop had evaporated (which occurred quite rapidly), another profile was recorded. The changes outside the scuff mark were much smaller than the changes inside. In either case most of the profile was lowered by the reaction.

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In another instance (fig. 3) the difference of reaction inside and outside the scuff mark was even more dramatic. Figure 3 presents a clear indication that changes of profiles can be both upward and downward (ie., surface material can be formed or removed). There is no doubt that the changes produced by the hydrochloric acid within the scuff mark were much greater.

Figure 4 shows the effect outside the scuff mark for two successive acid treatments on the bearing ball run to scuffing with polyphenyl ether (same surface as shown in fig. 3). Reactivity was greater for the second treatment than for the first. The first treatment may have attacked the oxide layer, while the second treatment may have attacked the metal itself.

**Interference Microscope Examinations of Uncoated Ball
Surfaces Run in Polyphenyl Ether Containing
1 Percent of 1,1,2-Trichloroethane (TCE)**

Figure 5 shows the profiles with a bearing ball in and outside the scuff mark obtained from the experiments with polyphenyl ether (5P4E) lubricant containing 1 percent of 1,1,2-trichloroethane (TCE). The profile differences produced by the hydrochloric acid were minimal, although the changes within the scuff mark were somewhat greater.

**Interference Microscope Examinations Of Titanium-Nitride-
Coated Bearing Ball Run in Polyphenyl Ether
with 1,1,2-Trichloroethane (TCE)**

Scuffing did not occur with the TiN-coated balls in the EHD experiments. Outside the barely visible score mark, the surface profiles did not change with the acid treatment; however, continuous profile changes occurred with acid treatment within the score mark. The reason for continuous changes was not understood; however, subsequent analysis with a scanning electron microscope (SEM) revealed a porosity in the TiN coating within the score mark (fig. 6(a)).

Figure 6(b) shows the integrity of the TiN coating outside the score mark. Presumably, these defects in the TiN coating within the score mark allowed the acid etch to penetrate through the coating and react with the subsurface.

**Surface Profiles of Previously Heated Steel
Plates Before and After Acid Treatment**

To answer the question whether prior heating to high temperatures would change the room temperature reactivity, the alcoholic hydrochloric acid solution was applied to previously heated steel specimens. Figure 7 shows profiles before and after treatment for one plate heated to 204° C and one to 774° C. In the former case the changes in contour were small, while in the latter case were rather large. Since the before and after profiles were not offset in this figure, the crosshatched area represents apparent surface growth due to acid treatment. Presumably this is due to corrosion products. Exactly how the surface chemistry and metallurgy are changed by preheating is not known. However, surface oxide formation from adsorbed oxygen is a likely possibility.

Nevertheless, it is obvious that preheating increases the surface reactivity toward hydrochloric acid.

An Arrhenius-type relation can be obtained by plotting the apparent surface profile changes (for a fixed planar distance) as a function of the reciprocal of the absolute preheating temperature (fig. 8). An apparent straight line function is obtained which indicates that a bearing surface preheated at one time to a high temperature will become more reactive at lower temperatures.

The Arrhenius plot of figure 8 indicates a lasting change in the metal surface by its preheating or tempering. Perhaps the so called chemostress coefficient defined by Ciftan and Saibe [6] plays a role. These authors have shown a change of chemical potential of an adsorbate with stress in the substrate. The chemical potential expresses the change of free energy with concentration of a chemical constituent. The free energy change (ΔG) for a reaction is related to the equilibrium constant (K) by the thermodynamic relation:

$$K = \exp (\Delta G/RT)$$

An Arrhenius relation between a product of reaction (e.g., surface profile change) and the absolute temperature can be derived by thermodynamic and continuum mechanics alone. However, the heterogeneity of the metal surface could be the basis of a kinetic explanation: Tempering, because it is diffusion-controlled, changes the surface composition logarithmically with temperature, and the chemical reaction rate depends on the number of reaction sites thereby created. This argument would also lead to an Arrhenius relation.

Both of these explanations could be valid, but the latter seems more important on the basis of chemical and, especially, electrochemical experiences. Their relative importance will be determined by repeating the experiments with pure materials.

SUMMARY OF RESULTS

The results of our experiments can be summarized as follows:

1. Scuffed steel bearing surfaces are more reactive toward alcoholic hydrochloric acid than unscuffed surfaces.
2. Scuffed bearing surfaces originally used with a lubricant containing a small concentration of an organic chloride are much less reactive toward alcoholic hydrochloric acid than surfaces scuffed with the base lubricant.
3. Titanium-nitride-coated (TiN) steel bearing surfaces did not scuff under conditions where the uncoated surfaces did. They did show score marks, however. Furthermore the TiN coating exhibited a porous surface structure in the score marks when run with polyphenyl ether containing 1 percent of 1,1,2-trichloroethane.
4. The reactivity of preheated metal surfaces toward alcoholic hydrochloric acid at room temperature follows an Arrhenius-type relation with temperature.

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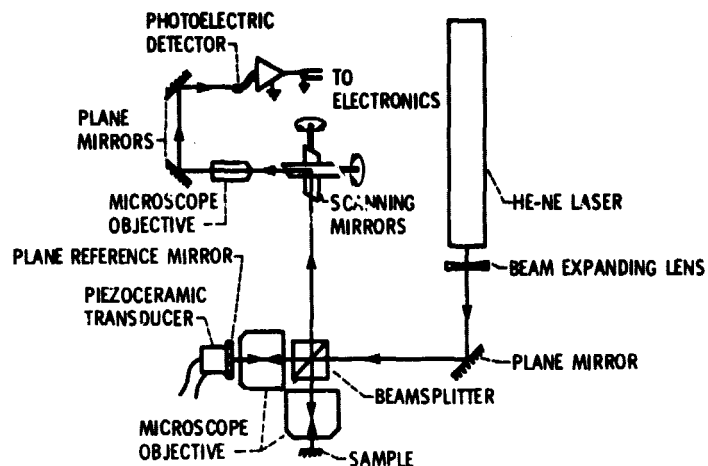


Figure 1. - Schematic drawing of interference microscope.

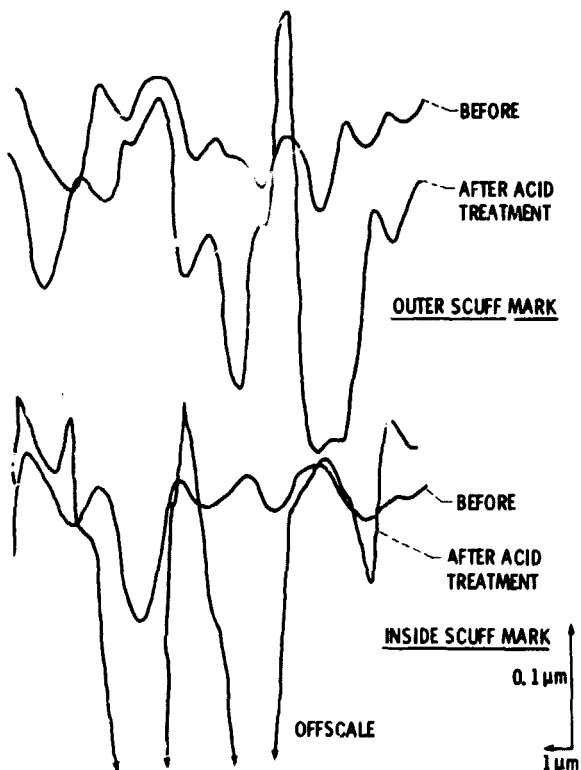


Figure 2. - Surface profiles of a stainless steel bearing ball scuffed in polyphenyl ether, before and after a drop of 0.04 M alcoholic hydrochloric acid was applied. (In order to avoid overlap, the traces taken after the acid treatment were offset.)

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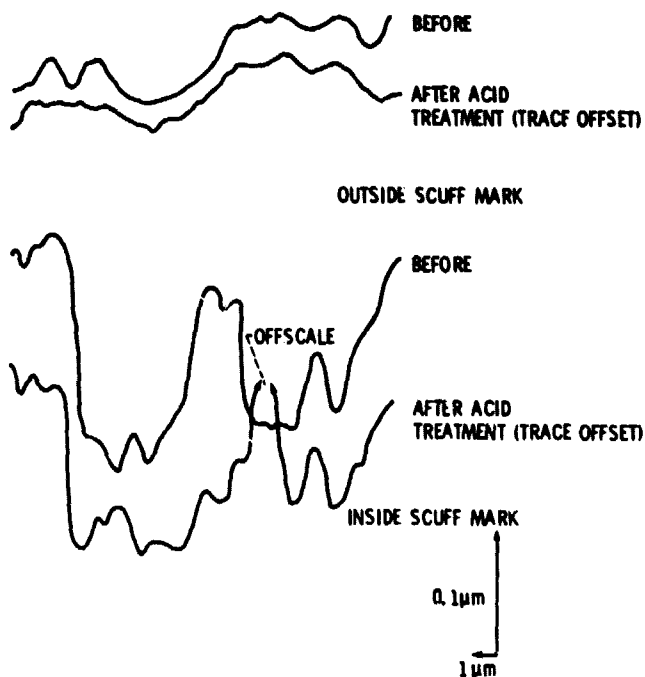


Figure 3 - Surface profiles of another stainless steel bearing ball scuffed in polyphenyl ether, before and after a drop of 0.04 M alcoholic hydrochloric acid was applied. (In order to avoid overlap, the traces taken after the acid treatment were offset.)

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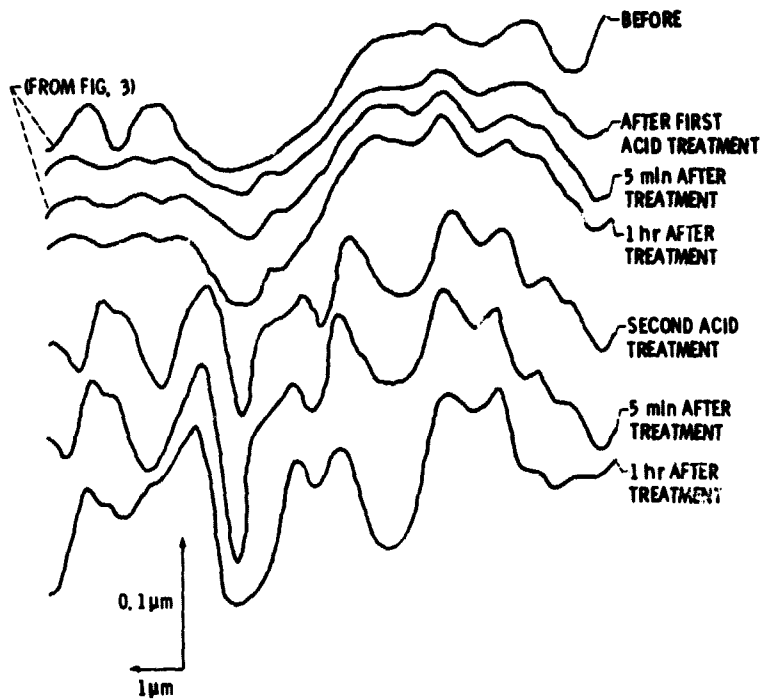


Figure 4. - Series of surface profiles of the bearing ball taken outside of the scuff mark. Two successive acid treatments were performed. (Profiles were offset to avoid overlap.)

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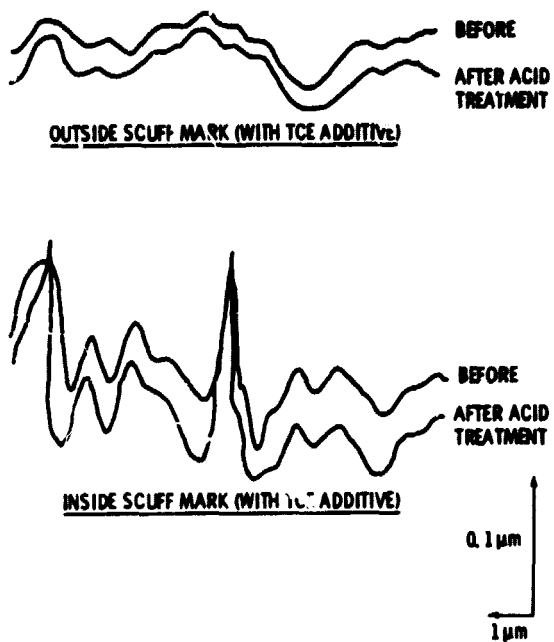


Figure 5. - Surface profiles of a bearing ball scuffed polyphenyl ether containing 1% of 1,1,2-trichloroethane. (The traces recorded after the acid treatment were offset to avoid overlap.)

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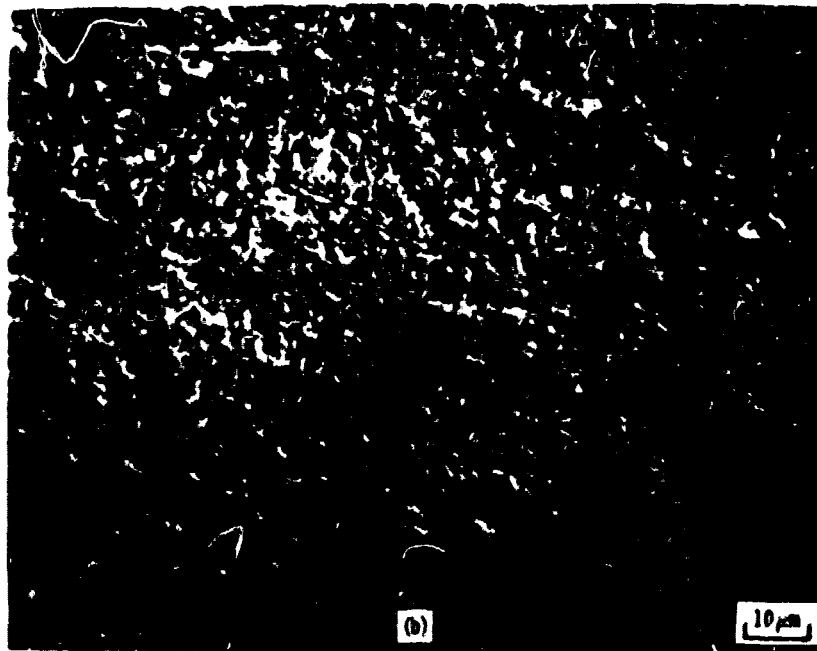
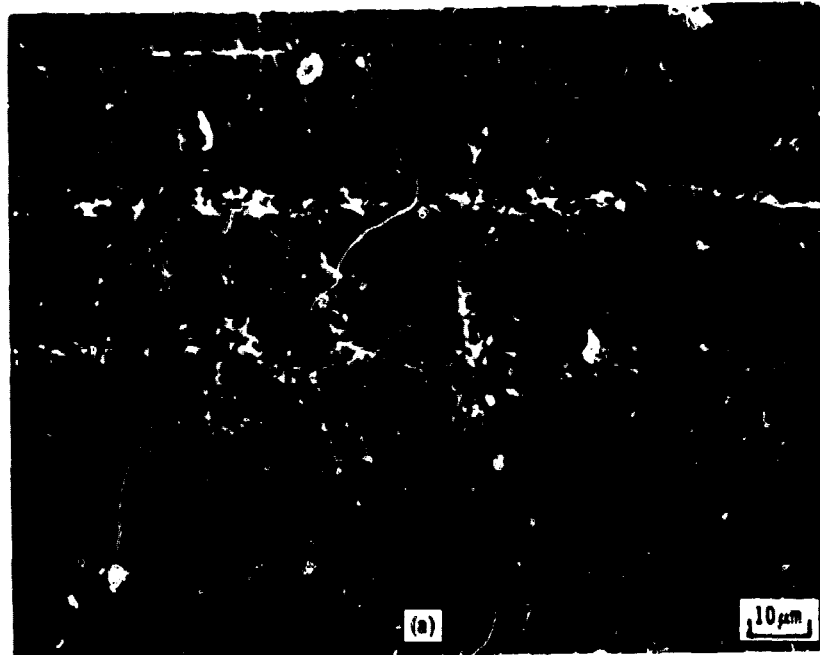


Figure 6. - Scanning electron micrograph of TiN-coated ball taken (a) within the score mark, note ladder-like porous structure. (b) outside the score mark, note porous structure. The lubricant was polyphenylether (SP4E) with 1% of 1,1,2-trichloroethane.

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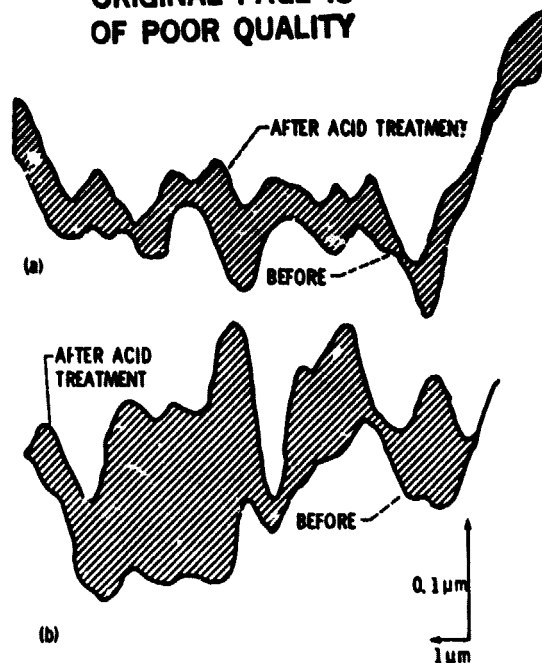


Figure 7. - Change of surface profile at room temperature after treatment with 0.04 M alcoholic hydrochloric acid of stainless steel specimen previously heated to (a) $T = 204^{\circ}\text{C}$, (b) $T = 774^{\circ}\text{C}$.

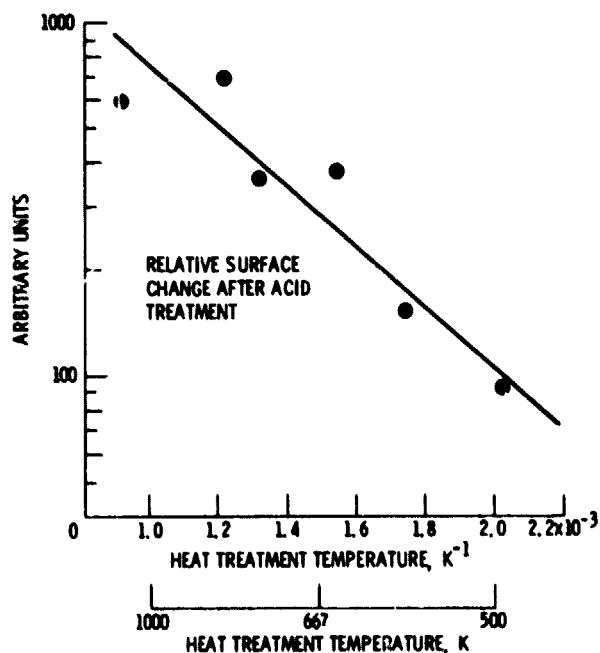


Figure 8. - Relative surface change after acid treatment of 304 stainless steel specimens previously heated to different temperatures.